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DMIC review OF RECENT DEVELOPMENTS

BDC

Corrosion and Compatibility

W. E. Berry • May 28, 1970

AD 869 538

ALUMINUM ALLOYS

General

The microbiological corrosion of aluminum alloys in jet fuel-water mixtures has been studied at General Dynamics.(1) Weight-loss data revealed the following percent increase in weight change of inoculated specimens over that of control specimens:

Alloy	Percent Increase
1100	71.4
2024	75.5
7075	184.2
7079	51.0

After 12 months' exposure, the maximum depth of penetration was found on the 2024 alloy, and the least penetration was in the 7079 alloy. The post-test results of examination are summarized in Table 1. The attack occurred as pitting, intergranular attack, and exfoliation. Fatigue tests were run on corroded and pitted specimens of each alloy, but the results were inconclusive when compared with fatigue results for either uncorroded specimens or those chemically corroded 10 mils.

Stress-Corrosion

A literature survey of recent European papers on the stress-corrosion cracking of aluminum alloys has been prepared by Battelle's Frankfurt Laboratories.(2) The survey covers mechanistic studies, new stress-corrosion tests, effects of cold work, and a general review of the problem. Also included in the survey are tables and graphs selected from the papers reviewed.

The exfoliation corrosion and stress-corrosion cracking behavior of several high-strength aluminum alloys has been studied at Alcoa as part of an overall program to determine fracture toughness, fatigue, and corrosion characteristics.(3) Resistance in salt spray, marine atmosphere, and industrial atmosphere was 7075-T73 ≥ X7080-T7, and X7080-T7 > 7075-T6 and 7178-T6. Stress-corrosion tests with precracked specimens (continuous immersion in 3.5 percent NaCl solution) and smooth tensile specimens (alternate immersion in 3.5 percent NaCl) rated the alloys and tempers in the same order. No failures were detected in any alloys stressed 75 percent of the yield strength in the longitudinal direction. Several 7075-T6 and 7178-T6 type samples showed some susceptibility to cracking at 75 percent of yield stress in the long transverse direction.

TABLE 1. MAXIMUM DEPTH OF PENETRATION OF VARIOUS CORRODED SPECIMENS

Corrosion Depth, mils	Number of Corroded Specimens 7075-T651	7079-T651	7178-T651	2024-T351
<1	57	75	73	26
1-5	21	12	23	14
5-10	9	3	15	32
10-15	2	0	4	14
15-20	0	0	2	1
20-40	0	0	0	1

TABLE 2. STRESS-CORROSION BEHAVIOR OF SAMPLES STRESSED IN THE SHORT-TRANSVERSE DIRECTION

Alloy and Temper	Resistance	Percentage of Yield Strength at Which Failures Were Observed	Failures Were Not Observed	Approximate K_{Iscc}, ksi
7075-T73510	Very high	--	75	≥20.5
X7080-T7E42	High	75	50	20.0
X7080-T7E41	Medium	34	25	--
7075-T6510	Low	15	--	≤12.0
7178-T651, T6510	Low	15	--	≤8.5

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The stress-corrosion behavior of samples stressed in the short-transverse direction is shown in Table 2.

A thermal-mechanical treatment for 7075 aluminum was developed by Rocketdyne that develops the yield strength of the -T6 treatment and the stress-corrosion-cracking resistance of the -T3 treatment.[4] Short-transverse yield strengths of 70 to 75 ksi and stress-corrosion lifetimes as long as those of 7075-T73 were achieved by an overage treatment of 7075-T6 for 5 to 10 hours at 350 F followed by a press-forging operation to give a 20 percent reduction in the longitudinal direction of grain orientation. These properties were obtained to the center of 3.125-inch-thick rolled stock and 4-inch-thick forged stock.

Australian scientists have studied the factors controlling stress-corrosion cracking in precipitation-hardened Al-5.7Zn-2.7Mg-1.35Cu alloy[5] Prenotched specimens were stressed by cantilever loading in the short-transverse grain direction, and a 0.5N NaCl/0.005N $NaHCO_3$ solution was allowed to drip continuously over the notch. Ninety-five percent of the life of the specimen was taken up by the formation of grain-boundary attack to a certain degree of acuity from which the stress-corrosion cracks emanated. The length of the incubation period was markedly influenced by stress level and aging treatment (metallurgical structure).

STEELS

Aqueous Corrosion

Means of protecting the nongrouted tendons of the prestressed concrete reactor vessel of the Fort St. Vrain Nuclear Generating Station have been investigated at Gulf General Atomic.[6,7] The steel wire conformed to ASTM Specification A421-65, Type BA. It was cold worked and thermalized (simultaneous stress and heat) to an ultimate tensile stress of 225 to 260 ksi. Marine-atmosphere exposure tests at LaJolla, California, indicated that a suitable combination of protective coatings and wraps would protect the tendons from rusting during fabrication, shipment, installation, and operation in the reactor. Studies with bare and coated notched specimens revealed that the tendons were not susceptible to stress-corrosion cracking or hydrogen-stress cracking. Irradiation tests with five organic systems revealed that the hydrogen generated as the result of radiolytic break down of the organic did not cause hydrogen-stress cracking of notched tendons stressed to 75 percent of the ultimate strength. Irradiation fluences were 1.3×10^{17} n/cm^2 (E > MeV) and gamma doses of 4.2×10^9 rads.

The effect of applied potential on the crack-growth rate of 180-ksi-yield-strength 10Ni-2Cr-1Mo-8Co steel in 3.5 percent NaCl has been studied by U. S. Steel.[8] Fatigue precracked cantilever specimens were used. Crack-growth was observed with both applied cathodic current and applied anodic current indicating that the alloy was susceptible to hydrogen cracking and stress-corrosion cracking. The mode of failure with no applied potential (at stress intensities above K_{Iscc}) could not be identified specifically as either hydrogen embrittlement or active-path stress corrosion.

U. S. Steel also has used applied potential to study the mechanisms of corrosion fatigue of 12Ni-5Cr-3Mo maraging steel solutions.[9] Wedge-opening loading specimens were fatigued at 600 and 6 cycles per minute. The results showed that the effectiveness of cathodic protection diminished as the crack front moved away from the free surface. The solution at the crack tip was pH 3, and localized regions of basic and acidic solutions were observed in the proximity of the central portion of the crack tip. Fractographic tests showed that hydrogen embrittlement was the primary mechanism responsible for acceleration of fatigue cracks in 12Ni-5Cr-3Mo steel in sodium chloride solution.

The corrosion-fatigue-crack growth rate of 4340 steel in distilled water has been studied at the University of Illinois by fracture mechanics techniques.[10] Tension-tension loads were applied to contoured, double cantilever-beam specimens. The results indicated that whenever the maximum cyclic stress intensity in the fatigue cycle was below the static threshold intensity for stress-corrosion cracking (K_{Iscc}), the environment no longer assisted the crack-growth rate. When the sustained (SCC) crack-growth rates approximated the corresponding fatigue crack-growth rates in air, the effect of high frequencies and high-stress intensities was to decrease the fatigue-crack-growth rate to less than the growth rate of the stress-corrosion crack. Suggested reasons for this behavior were a chemical change in the crack-tip environment or strain rate dependence of the plastic deformation at the crack tip.

Hydrogen Embrittlement

Studies on the embrittlement of 4130 steel in low-pressure, gaseous hydrogen have been reported by NASA - Ames Research Center.[11] High-strength 4130 steel was embrittled by straining in gaseous hydrogen at pressures less than 1 atm. The embrittlement was caused by hydrogen-induced slow crack growth. The comparison of the characterisitics of gaseous-hydrogen embrittlement with those of embrittlement of hydrogen-charged steels suggested that both types of embrittlement were basically the same phenomenon. The proposed mechanism of embrittlement involved a surface reaction between the hydrogen and the steel which facilitated crack propagation by either lowering the surface energy or changing the energy of plastic deformation.

A generalized model for hydrogen embrittlement of steel has been proposed by Argonne National Laboratory scientists.[12] The model proposes that hydrogen embrittlement does not occur until a hydrogen-rich phase has been precipitated from solution, and the precipitation of this phase can be induced by triaxial tensile stresses. Considered were the effects of substructure (including prismatic dislocation loops), test temperature, cooling rates, solubility, diffusivity, and notch-sensitivity on ductility. Two of the more popular models for hydrogen embrittlement were shown to have salient features that were compatible with the proposed generalized model.

Lead Embrittlement

The lead-embrittlement of steel has been studied by Illinois Institute of Technology.[13] Embrittlement occurred when the lead was present either internally in the steel (leaded steel) or soldered to the surface. Embrittlement occurred over a temperature range of 300 F (more than 300 F below the melting point of lead) to a brittle-to-ductile transition temperature of 700 to 900 F. Embrittlement was more severe in steels with low-carbon or low-alloy content. Lead content in a leaded steel was unimportant, but the compositio.. of the lead was critical. Additions of antimony, arsenic, tin, or zinc to lead increased the severity of embrittlement at temperatures below the melting point of lead, and increased the brittle-to-ductile transition temperature by 200 F. Decreasing the grain size had no effect at temperautres below the melting point of lead; but, at higher temperatures, it lowered the transition temperature. Increasing the loading rate increased the ductility markedly at temperatures below the melting point of lead, while, at higher temperatures, lowering the loading rates lowered the brittle-to-ductile transition temperature. The low-cycle fatigue life of leaded steel was unchanged relative to that of nonleaded steel at 300 F, but was reduced by about one order of magnitude at 600 F. The results of the study were interpreted to indicate that at temperatures below the melting point of lead, the embrittlement is controlled by the rate of transport of lead atoms to the tip of propagating subcritical cracks, while at temperatures above the melting point, fracture is nucleation controlled, and, once a crack nucleates, it grows rapidly.

STAINLESS STEELS

Precipitation-Hardening Stainless Steels

The stress-corrosion-cracking behavior of precipitation-hardening stainless steels has been studied at the George C. Marshall Space Flight Center.(14) Bent beams, loaded tensile specimens, and C-rings were used to test the materials in the longitudinal and transverse directions of grain orientation. The stressed specimens were alternately immersed (10 minutes) in 3.5 percent NaCl solution and air dried (50 minutes) at room temperature. The results indicated that PH13-8Mo, PH14-8Mo, 15-5PH, A-286, Almar 362 and Unitemp 212 stainless steels were resistant to stress-corrosion cracking under these conditions in all heat treatments studied. The PH15-7Mo and 17-7PH alloys were susceptible to stress-corrosion cracking in all conditions studied except 17-7PH - CH 900. The 17-4PH - H 900 treated specimen cracked in the transverse grain-direction. AM-350 alloy was susceptible to cracking in the SCT 850 condition, but not in the SCT 1000 condition.

The cathodic protection of stressed 17Cr-4Ni-4Cu precipitation-hardened steel in seawater has been studied by the Naval Ship and Development Laboratory.(15) Specimens were aged at 900 and 1050 F (175 and 154-ksi yield strength) prior to testing as-bent beams at 90 percent of yield strength. Cracking occurred in both the 900 and 1050 F aged specimens that were not cathodically protected. Applied potentials of -160mv to SCE prevented stress-corrosion cracking in both aged conditions, but did not prevent crevice corrosion. An applied potential of -1375mv to SCE caused cracking of the 900 F aged specimen (presumably from hydrogen embrittlement), but prevented cracking and crevice corrosion of the 1050 F aged specimen.

Austenitic Stainless Steels

The stress-corrosion cracking of U-bends of Type 304 stainless steel in 5N H_2SO_4 + 0.5N NaCl at room temperature has been studied by scanning electron microscopy in England.(16) Specimens were solution annealed at 1050 and 1300 C (1920 and 2370 F) or were cold worked 11 and 33 percent. Cracking times ranged from 4 to 21 days, and calculated corrosion rates ranged from 10 to 117 mils/year. In general, cracks initiated intergranularly, but propagated in a transgranular fashion. Crack propagation occurred as the result of the formation of parallel tunnels across a grain. Cracking was believed to result from the interaction of dislocation pile ups and the environment. The pile ups, occuring on slip planes, account for the transgranular mode of crack propagation.

The effect of alloy additions on the stress-corrosion cracking of iron-chromium-nickel alloys in boiling $MgCl_2$ solutions has been studied at The Ohio State University.(17) Nickel additions in the 5 to 15 percent range produced the usual maximum susceptibility to stress-corrosion cracking. Chromium additions in the range of 10 to 15 percent to binary iron-nickel alloys produced maximum resistance to cracking. Alloys containing 10 to 15 percent chromium and 10 to 15 percent nickel plus small additions of aluminum, beryllium, and copper were particularly resistant. Of the fourth component additions to a Fe-20Cr-15Ni alloy, the elements of the platinum group (platnium, iridium, osmium, rhodium, ruthenium, palladium), group VA (nitrogen, phorphorus, arsenic, antimony, bismuth), and molybdenum produced the most deleterious effects. The most beneficial additions were aluminum, beryllium, carbon, silicon, and possibly boron.

A review of the literature on the stress-corrosion cracking of iron, nickel, chromium, and their alloys in caustic solutions has been prepared by The Ohio State University.(18) The review was prepared in support of the liquid-metal cooled, fast-breeder reactor program in considering an accident or leak that would permit sodium to oxidize or hydrolyze to produce $NaOH$-Na_2O-H_2O solutions. Accordingly, the environment considered was primarily $NaOH$-H_2O over a broad temperature range. Information is presented in areas of thermodynamics, electrochemical kinetics, corrosion rates, stress-corrosion-cracking phenomena, structures of passive films, and inhibitors.

NICKEL- AND COBALT-BASE ALLOYS

The corrosion behavior of Multiphase Alloy MP35N (35Ni-35Co-20Cr-10Mo) has been studied by Standard Pressed Steel.(19) The alloy exhibited good-to-excellent corrosion resistance at 50 C (122 F) in 10 and 78 percent H_2SO_4, 10 and 65 percent HNO_3, 10 percent NaCl (pH 2 with HCl), 10 percent $FeCl_3$, and 10 percent HCl plus 1 percent $FeCl_3$ (but not in 10 percent HCl alone). U-bends of the alloy did not crack after 192 hours in boiling 42 percent $MgCl_2$. The alloy is an extremely noble metal with a potential of +0.056 (versus Ag-AgCl) in seawater. It is cathodic to most materials and caused galvanic corrosion of K-Monel, Type 316 stainless steel, and carbon steel in couple tests conducted in seawater. No galvanic corrosion was noted in similar tests conducted with titanium-MP35N couples. Wire-rope samples made of MP35N alloy have shown no evidence of attack after 2.5 years' exposure in the mud, seawater, tidal zone, splash zone, or marine atmosphere.

TITANIUM ALLOYS

The second annual report on the mechanism of stress-corrosion cracking of titanium alloys in N_2O_4 and aqueous and hot-salt environments has been issued by Battelle's-Columbus Laboratories.[20] The mechanism of aqueous stress-corrosion cracking most consistent with the experimental observations was related to the formation of strain-induced hydrides in the active [10$\bar{1}$0] slip bands, which inhibited plastic flow around the crack tip and, thereby, promoted cleavage. The mechanism of stress-corrosion cracking in pure N_2O_4 was related to oxygen which acted as a cathodic depolarizer. That is, titanium reacts with N_2O_4 to form NO^+, NO_2^+ and unstable nonprotective $TiO(NO_3)_2$ (anodic reaction). Oxygen reacts with N_2O_4 (cathodic reaction outside the crack) and NO^+ and NO_2^+ (cathodic reaction inside the crack). The unstable $TiO(NO_3)_2$ decomposes to TiO_2, N_2O_4, and O_2, and the latter helps to depolarize cathodic reactions within the crack. Results with hot salt were inconclusive and these studies are being repeated.

The stress-corrosion cracking of titanium and Ti-5Al-2.5Sn alloy in methanol-chloride has been examined by scanning electron microscopy in England.[21] U-bends and dynamic tensile straining were used. The alloy exhibited transgranular cleavage; and the mode of cracking in unalloyed titanium changed from intergranular separation to transgranular cleavage as the impurity levels were increased in the titanium. Small amounts of hydrogen introduced into the most impure titanium resulted in a larger amount of cleavage in subsequent stress-corrosion tests and, in air, fractures that were similar to stress-corrosion fractures. The results were interpeted in terms of intergranular dissolution or as arising mainly from impurity segregation and transgranular cleavage due to a form of hydrogen embrittlement.

The influence of salt and elevated-temperature exposure on the maximum compressive strength of titanium-alloy skin-stringer panels has been investigated by the NASA/Langley Research Center.[22] Ti-8Al-1Mo-1V specimens were tested to failure at room temperature after being coated with a 3.4 percent NaCl solution and exposed 1000 hours at 600 F to develop stress corrosion cracks. The hot-salt stress-corrosion cracks had little effect on the compressive strength of the panels, although considerable tearing and deformation appeared to initiate from the cracks at maximum load.

BRAZE ALLOYS

The compatibility of brazed joints with N_2O_4 has been studied by the Air Force Rocket Propulsion Laboratory.[23] Lap-joint panels of Type 347 stainless steel were prepared with each of the following braze alloys: 82Au-18Ni-0.3Li, 70Au-22Ni-8Pd, and 72Ag-28Cu-0.2Li (weight percents). Stressed and unstressed specimens were exposed 360 days to the liquid, vapor, and liquid-vapor interface of N_2O_4 with 0.2 percent water (in conformance with MIL-P-26539A). Some surface corrosion was detected on all alloys after exposure, but there was no evidence of stress-corrosion cracking or galvanic attack. It was concluded that these braze alloys were acceptable for use with N_2O_4 systems.

COATINGS

The reasons for accelerated attack caused by one particular antifouling paint have been studied by the Naval Applied Science Laboratory.[24] The paint in question was different from other antifouling paints studied in that it had copper-metal pigment in its formulation and had a lower electrical resistance, and it was the only coating to exhibit a potential in seawater (-0.15 volt versus SCE). Circular holidays 1/8 in. in diameter produced pit depths in the underlying steel of up to 47 mils in 2 months' exposure in aerated "sea salt" in the copper-metal-pigmented paint and less than one-half that value in the other antifouling formulations.

Treatments for aluminum aircraft surfaces which confer corrosion resistance and enhance the adhesion of military-specification epoxy paints have been described in a final report prepared by American Cyanamid Company.[25] The experimental approach was on the basis that selected chelating agents would chemically bond to the surface and, if appropriately substituted with epoxide reactive groups, would also tie into the paint coating. Most of the evaluation was based on salt-spray exposure of painted panels followed by an adhesive peel test. Two agents were found to be promising: dimethylphosphonoacetamide (DMPA) and 1-amino-2 phenylethyl phosphonic acid (APEPA); the DMPA was favored because of its greater water solubility. The optimum formulation consisted of a 3-minute (or longer treatment) with a pH 3 aqueous solution containing 1 to 2 weight percent DMPA. The shelf life of DMPA-treated panels was acceptable on the basis of extended atmospheric exposures of treated samples which produced only a slight loss in effectiveness. The performance of the DMPA treatment was equal to or slightly better than that of conventional chromate pretreatments and, in addition, the DMPA required no surface cleaning, was cheaper, was not restricted by a critical application time, and had better shelf life. Preliminary tests indicated that the DMPA and APEPA were suitable treatments for promoting adhesion of paint to titanium.

The results of a study to develop aerodynamically smooth corrosion-protective systems for fastener-head countersinks in highly loaded aircraft skins have been presented in a final report issued by Vought Aeronautics.[26] Corrosion tests in acetic acid salt spray and at Kure Beach showed that a polyurethane elastomer was protective after simulating limit loads of A-7 wing fold areas. Polysulfide and epoxy films cracked during loading and a silicone material was easily abraded and not protective. Permeability measurements showed the polyurethane was protective in films as thin as one mil in the peripheral groove around the fastener. The polyurethane elastomers yellowed in the sunshine, but a linear-polyurethane nonyellowing coating could be used as a top-coat material to form a durable flexible system.

GENERAL

A manual on Corrosion Control for Manned Space Flight Network Facilities has been prepared by

Bendix Field Engineering Corporation for the Goddard Space Flight Center.[27] The manual discusses the various forms of corrosion, the corrosion resistance of aluminum-, iron-, magnesium-, and nickel-base alloys, the behavior of metal and organic coatings, and the methods of corrosion control.

A chemical engineer's guide to seawater has been prepared by the Dow Chemical Company.[28] Information in the papers on seawater covers average monthly temperature and salinity at various locations off the U. S. coast, chemical composition, physical properties at concentrations varying from that of fresh water to four times normal seawater, foaming and turbidity characteristics, deposition of solids, and marine life.

The ARPA Coupling Program on Stress-Corrosion Cracking has compiled abstracts from journal articles, recent reports, and talks generated under this program.[29] The abstracts cover aluminum alloys, high-strength steels, high-strength stainless steels, and titanium alloys, as well as fundamental studies on the mechanisms of stress-corrosion cracking. Also included are selected abstracts from outside the ARPA program in the field of stress-corrosion cracking.

The role of oxide plasticity on the oxidation behavior of metals is reviewed in a paper by Denglass of the University of California at Los Angeles.[30] Included in the discussion are the structural aspects of the scale; the effects of stoichiometry, temperature, and film thickness on plasticity of oxides; causes of micro- and macrostresses in the oxide film; measurement of stresses in the oxide; and film spalling.

In-line corrosion probes to detect and measure galvanic and crevice corrosion in seawater have been developed by Battelle Northwest.[31] The probes were evaluated in ambient seawater, artificial seawater at 100 C, (212 F) and artificial seawater in a pilot plant at 150 C (302 F) and 20 ft/sec. The steel probes developed an adherent Fe_3O_4 film and corroded at low rates (about 1 mil/year) at 150 C. This film afforded short-term protection when the probe was transferred to saline water at a lower temperature; but, eventually the film broke down and the specimen corroded at a higher rate more characteristic of those for bare specimens at the lower temperature. Crevice probes made with 6061 aluminum and exposed at 150 C revealed an initial low rate (incubation period) before the onset of crevice corrosion. The indicated corrosion rate during crevice attack determined by measuring the diameter of the wire in the area of greatest attack was about 500 mils/year compared with a calculated corrosion rate of about 900 mils/year. The measured attack appeared to be less severe because the resistance probe tends to measure an average value over the entire probe.

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